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TECHNICAL REPORT BRL-TR-2596

EVALUATION OF BLACK POWDER PRODUCED BY
INDIANA ARMY AMMUNITION PLANT

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) shg Closed-bomb techniques were used to determine quickness and burning-rate equations for two standard class-one black powders made by DuPont and GOEX; these results were compared to similar measurements on material produced by the Indiana Army Ammunition Plant. The differences in burning-rate exponents derived from closed-bomb and strand burn-rate experiments are discussed in terms of modes of combustion. Other comparisons were made between the black powders using flame-spread rate data obtained in 105mm Howitzer primer tubes		

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20. Abstract (Cont'd):

both plugged and unplugged. Other parameters investigated include: free volumes, strand-burn rates, true and bulk densities, and grain-size distributions. It is suggested that black powder produced by the Indiana Army Ammunition Plant is becoming, or has become, the most extensively characterized lot of black powder produced in the United States.

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I. INTRODUCTION

The U.S. Army Corps of Engineers built a continuous black powder manufacturing facility in Charlestown, IN. The equipment was installed and operated by ICI Americas, Inc. (ICIA). The plant, a GOCO facility, incorporates a jet-mill for grinding material to about fifteen microns and follows the Norwegian design of Kjell Lovold.¹ The Indiana Army Ammunition Plant (INAAP) produced its first "prove-out" batch of black powder, about 1000 pounds, in March 1983 and the production process will be described in an ICIA report.² In contrast, our report evaluates INAAP black powder in relation to its combustion properties by closed-bomb and flame-spread techniques. Other tests are planned by the ARDC Product Assurance Directorate (DRSMC-QAR-R), to include an extensive gun-firing program, to be conducted at the Jefferson Proving Ground, IN, funded by an Engineering Study.* The ultimate question addressed by all of this testing is to determine if the new production facility has produced black powder that can perform properly in a gun environment and, also, to devise laboratory tests to predict this property.

One main purpose of this evaluation study, other than to characterize black powder, was to compare the flame-spread rates as measured in the flame-spread tester to other combustion data to determine if the flame-spread tester could be used as a quality assurance device to ascertain "good or bad" black powder as defined by gun-firing performance.

Even though black powder is the oldest known propellant, little testing has been recorded under the same conditions and this lack of a published data base hinders evaluation. Current work is attempting to bridge this gap. Closed-bomb testing was employed and the work was sponsored by the Munitions Production Base Modernization Agency, MPBMA prove out project in support of project 5812084. This approach is but one tool to evaluate combustion and with this technique we hoped to define similarities and differences between two Indiana lots and the DuPont-GOEX black powders chosen as reference standards. As part of the production trial the products of two suppliers of potassium nitrate were used, one being a non-neutral material from Vertac Chemical Corp. of Vicksburg, MS, and a neutral salt from Croton Chemical Co.

¹Kjell Lovold, "Process For the Preparation of Black Powder," U.S. Patent No. 3660546, 2 May 1972.

²ICI Americas, Inc., "Black Powder MFG. Plant - Phase II," Vol. 1-5, Indiana Army Ammunition Plant, Charlestown, IN, 1984.

*Sponsored by DRSMC-LE(R), AMCCOM project no.; ESP 1A-3-8428. They also funded flame-spread tests.

of South Plainfield, NJ. Some previous testing of these two salts was supported and reported by the Product Assurance Directorate, Dover, NJ.³

Other limited data are included such as flame-spread rates, density, grain-size distributions, and strand-burn rates. Every attempt was made to characterize, as fully as possible, black powder made by the Army Ammunition Plant (INAAP).

II. EXPERIMENTAL

A. Grain-Size Distribution

Class-one black powder consists of grains that pass through a number four sieve (4.75mm) and not a number eight sieve (2.36mm). One hundred grams of each of the black powders was placed on a stack of sieves, four through eight, and the assembly was shaken. The portion held by each sieve was weighed.

B. Density

The bulk density of black powder is normally given as the ratio of its weight divided by its volume as measured by the amount of mercury displaced by the sample. Such values were measured by ICI Americas, Inc., and are given in this report. Another type of density, true density, can be obtained by estimating the sample volume by measuring the amount of helium displaced by the sample. The technique does not include pore volume and this density is the maximum theoretical density of the sample. From these two densities the internal free-volume can be estimated. Previous work used ethyl ether for this purpose. The helium "AutoPycnometer" was made by Micromeritics of Norcross, GA and the measurements were performed by Robin J. Davals of BRL-TBD, Aberdeen, MD.

C. Flame-Spread Rates

Open air flame-spread rates were obtained by measuring the time required to burn 16g of black powder strung out in a straight line 46cm long. A recording TV camera was used to measure burn times. Examples are given by White, Holmes, and Kelso.⁴ The technique was improved by placing the grains on a plastic strip and using a mirror to photograph the under-side of the silhouetted burning string. This placed the black powder between the camera and the light.

³(a) A. Smetana and H. Gults, "The Effects of the Use of Non-Specification Grade Potassium Nitrate in Black Powder," QAR-R-006, Product Assurance Directorate, Dover, NJ, September 1982. (b) A. Smetana and H. Gults, "Effects of Non-Specification Grade Potassium Nitrate in Black Powder," ARPAD-TR-83001, Product Assurance Directorate, Dover, NJ, May 1983. AD-E401-002.

⁴K.J. White, H.E. Holmes and J.R. Kelso, "Effect of Black Powder Combustion on High and Low Pressure Igniter Systems," Proceedings of the 16th JANNAF Combustion Meeting, CPIA publication 308, Vol. 1, p. 477, September 1979.

Semi-confined flame-spread rates were obtained and the test fixture was modeled from the high-pressure bayonet type primer (M28B2) used in the 105mm Howitzer. The tube is 26cm long and its inside diameter is 1.15cm. The tube is drilled with four columns of 11 holes, each of 0.33cm diameter. As received, the holes are plugged with a "wax-like" substance. The distance between detector ports was chosen as the maximum possible and was 19cm. The tube was filled with 19.4g of black powder.

Burning times have been measured in these tubes by different authors several ways that include: high-speed photography, front and rear pressure gauges, and front and rear light detecting diodes. Confined tests have been performed with the holes plugged and brisant-ignition (M16) primers have been used as well as soft ignition employing electric matches. Soft-ignition has also been applied to tubes where the holes were open. In general, the plugged assembly generates 1500psi and the tube with the wax removed from the holes generates 500psi.

D. Closed-Bomb Evaluation

Closed-chamber experiments were performed in a Technoproducts Impulse Bomb which has an internal volume of 88.5 cubic centimeters. Class-one black powder was evaluated at a loading density of 0.14 gram per cubic centimeter. To achieve a better degree of reproducibility, the charges were placed in Dacron bags with an Atlas M100 electric match inserted in the center of the charge. Pressure was measured with a Kistler 607C3 pressure transducer and Kistler 504E charge amplifier. Data were acquired and recorded using a Nicolet Explorer III digital oscilloscope. Data reduction was performed on a PDP 11/34 minicomputer using the CBRED2 program. Grains were considered to be perfect spheres with a diameter chosen at the midpoint of the size range of the original screening and a value of 0.14 inch was assumed. The GOEX and DuPont data were reduced using the same thermochemical values and the Indiana samples were treated independently due to slightly different chemical compositions. All of the chemical compositions are given in Table 1.

Closed-bomb techniques and the subsequent data reduction have been a concern of several laboratories. To insure equivalent data processing, many laboratories exchanged propellant samples and the results are given in a JANNAF Combustion report.⁵ Although black powder was not considered, this document does reflect the BRL and other laboratories' closed-bomb evaluation methods and the description is a valuable reference tool.

The input to the thermodynamic codes requires the chemical composition of the propellant and in the case of black powder, the codes require the composition of charcoal as well as the other ingredients. Eli Freedman⁶

⁵JANNAF Combustion Subcommittee, Burn Rate Measurements and Data Reduction Procedures Panel. "Round Robin Results of the Closed Bomb and Strand Burner," A. Juhasz, Ed., CPIA Publication 361, July 1982.

⁶Eli Freedman, "The Thermodynamics of Real and Unreal Black Powder," Proceedings of the 20th JANNAF Combustion Meeting, CPIA Publication 383, Vol. 1, p. 511, October 1983.

TABLE 1. Chemical Composition of Black Powders

DuPont 111-12 and GOEX 75-44		IND-83B 001-003		IND-83B 001-004	
	KNO ₃	73.80	KNO ₃	72.93	74.61
	S	10.26	S	9.66	9.76
C _{8.68} H _{4.96} O _{1.00}	Charcoal	15.54	Charcoal	15.97	15.24
CaCO ₃	Ash*	0.10	CaCO ₃	0.99	0.00
	H ₂ O	0.30	H ₂ O	0.46	0.39

*To simplify calculation ash was assumed to be just CaCO₃
Values are in weight percent

performed such calculations for the Indiana, GOEX, and DuPont samples evaluated in his and this report. For the first time, charcoal has been represented by its elemental composition as opposed to only carbon as had been the custom. For the GOEX and DuPont samples, Freedman used the chemical composition of charcoal given by Rose⁷ as "Roseville B" made by the Roseville Charcoal Co. of Zanesville, OH, and the potassium nitrate/sulfur/charcoal concentrations were taken from the data sheets supplied by Indiana. In a like manner, the chemical composition of the charcoal used at Indiana (also made by Roseville) was taken by averaging the ten values reported by Sasse⁸ and again, potassium nitrate/sulfur/charcoal concentrations were obtained from Indiana data sheets. The values used are given in Table 1.

III. RESULTS

A. Closed-Bomb

DuPont and GOEX standards and two Indiana lots were burned in the closed bomb. Four samples of each type were evaluated and burning-rate curves are given as a function of pressure in Figures 1, 2, 3, and 4. The GOEX data set, Figure 2, shows two distinct and different curvatures which also appears in one DuPont curve, Figure 1. We have no explanation for this abnormality. For comparison one curve from each data set, the one with the highest ordinate values, is given in Figure 5. This selection was made to present the worst case; however, an average curve could have been chosen or the curves derived from the burning rate equations could have been shown that have, in themselves, an averaging effect in that data is put in a standard form. One author, R.S., prefers overlays to perceive differences between data sets. For closer examination, selected burn rates, at 500psi intervals, are given in the appendix for all of the closed-bomb experiments.

Table 2 contains the corresponding values of computer input data and results of the thermochemical calculations. The important parameters of quickness and burning rate are given in Table 3. Taking the average quickness from each data set and taking GOEX 75-44 as the reference standard the relative quickness of DuPont 111-12 is 76.1%, Indiana-003 is 64.0% and Indiana-004 is 61.8%.

The burning-rate curves of GOEX black powder, Figure 2, show poor reproducibility in the low pressure region. The curves break at approximately 1500psi for two of the three samples in the data set. Two samples exhibit smoother burn-rate curves throughout the entire pressure range. Above 2000psi, the GOEX samples show good reproducibility. The DuPont samples, Figure 1, exhibit some similarity to the GOEX material in that one curve shows a break at 1500psi and again there is poor reproducibility in the low pressure

⁷J.E. Rose, "Investigation on Black Powder and Charcoal," IHTR-433, Naval Ordnance Station, Indian Head, MD, September 1975.

⁸R.A. Sasse', "Characterization of Maple Charcoal Used to Make Black Powder," ARBRL-MR-03322, Ballistic Research Laboratory, Aberdeen, MD, November 1983, ADA-136-513.

TABLE 2. Thermodynamic Calculations for Black Powder

	<u>GOEX 75-44</u>	<u>DuPont 111-12</u>	<u>IND-003</u>	<u>IND-004</u>
Weight (g):	12.6280	12.6660	12.6450	12.7390
Density (g/cm ³):	1.76	1.79	1.77	1.79
Initial Temperature (Deg K):	294.	294	294	294
Theoretical Impe... (Ft-lb/lb):	95583	95583	93056	96417
Flame Temperature: (Deg K)	2038	2038	1822	1945
Average Molecular Weight of Products	59.25	59.25	54.41	56.06
Co-Volume (Cubic Inch/lb):	27.670	27.670	27.000	27.200
Gamma (Ratio of Specific Heats)	1.269	1.269	1.222	1.214
Theoretical Max Pressure (kpsia):	6.940	6.964	6.740	7.051
Surface Area (Square Inches):	18.756	18.497	18.675	18.600
Number of Grains:	304.6	300.4	303.3	303.1

region. This is also true when comparing the DuPont and GOEX curves. Both Indiana lots, Figures 3 and 4, burning-rate curves are more linear than the DuPont and GOEX data. Indiana lot 3 showed the best overall reproducibility and GOEX samples burned fastest. The two Indiana lots burned in similar fashion and had values near, but slightly below, the DuPont samples.

Quickness values are averages of values taken at 0.250, 0.375, 0.500, and 0.650 of P/P (max) and they are given in Table 3 with the coefficients for the

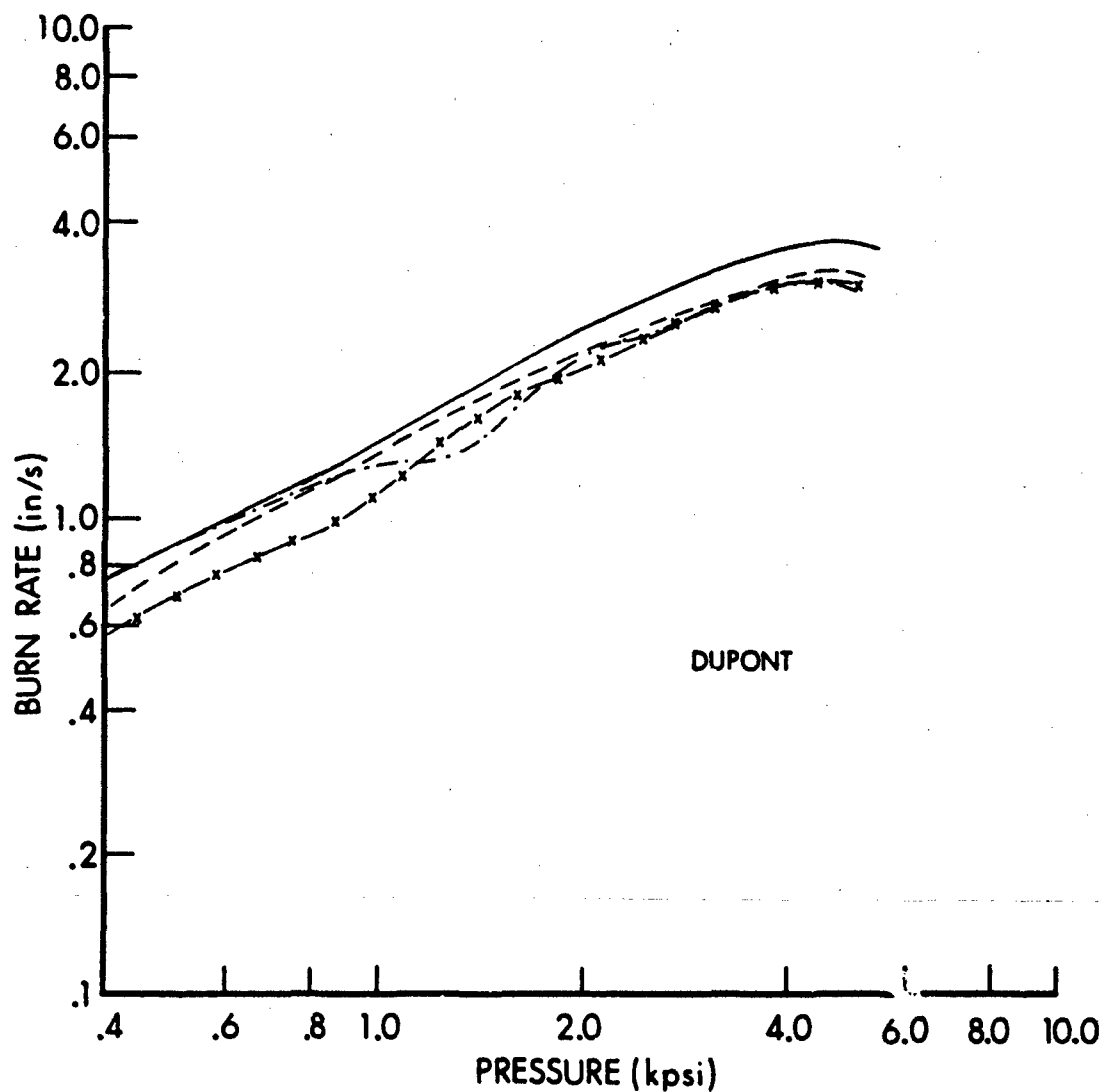


Figure 1. Burning-Rate of DuPont 111-12 Black Powder as a Function of Pressure

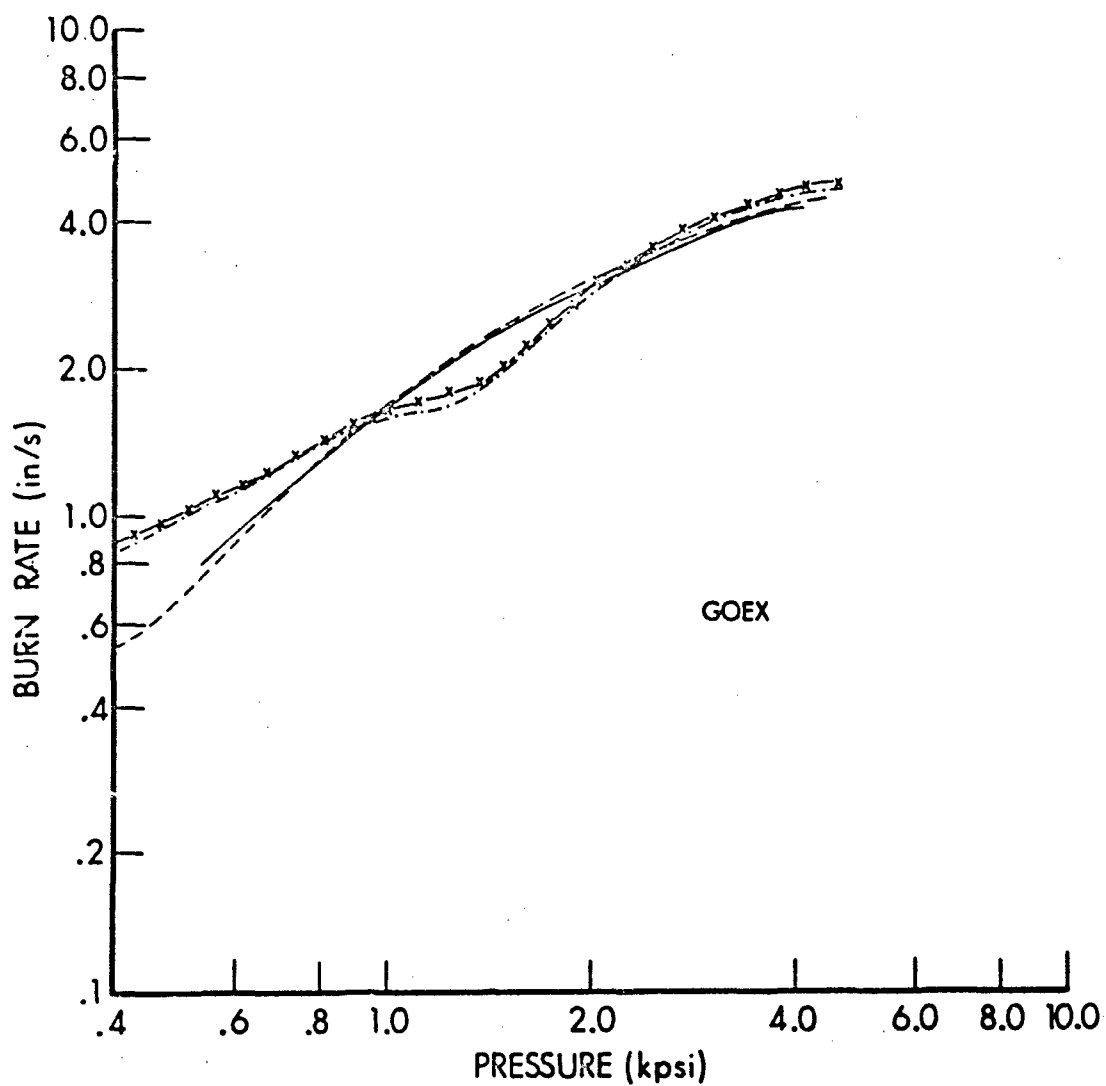


Figure 2. Burning-Rate of GOEX 75-44 Black Powder as a Function of Pressure

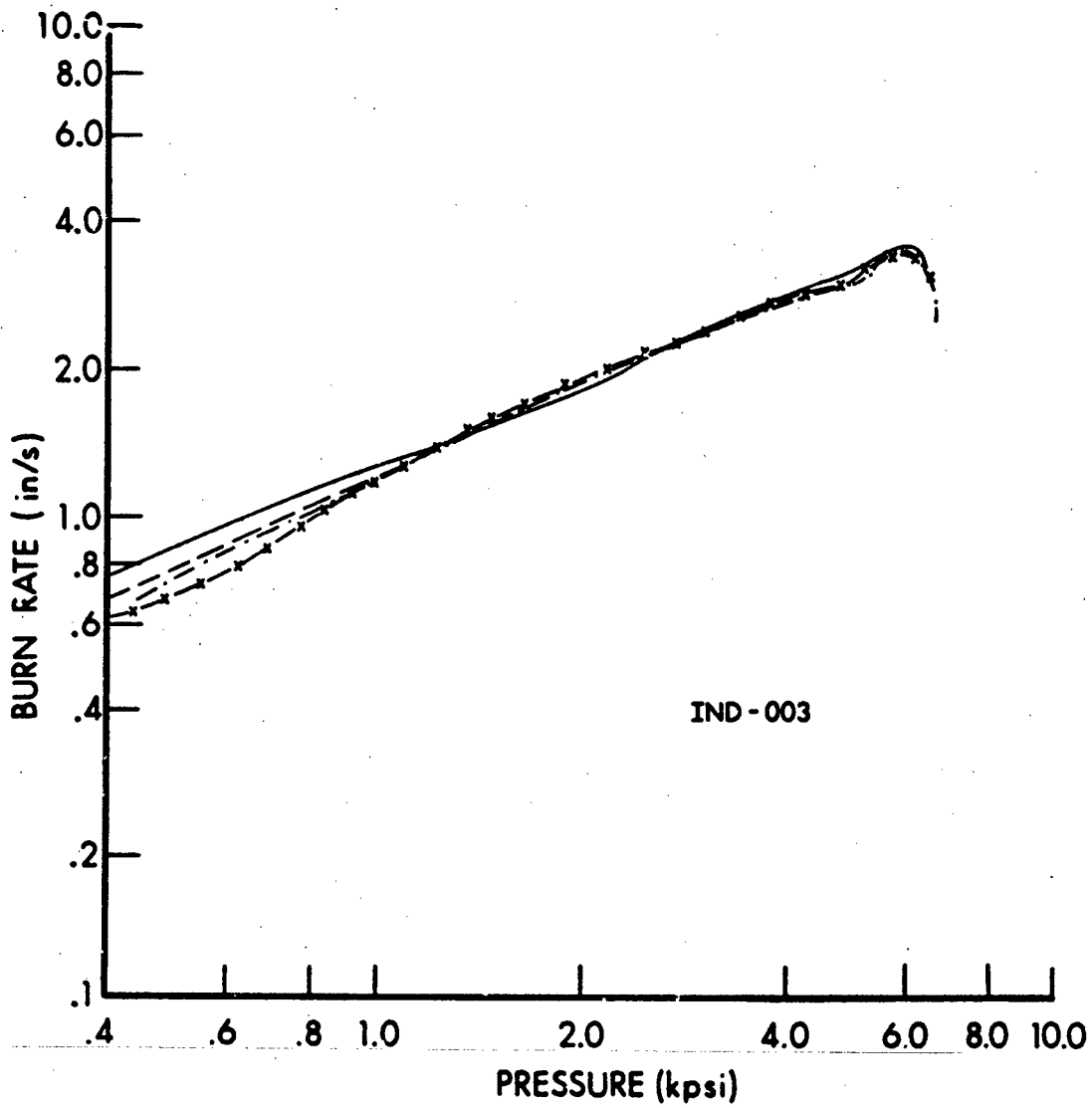


Figure 3. Burning-Rate of IND-003 Black Powder as a Function of Pressure

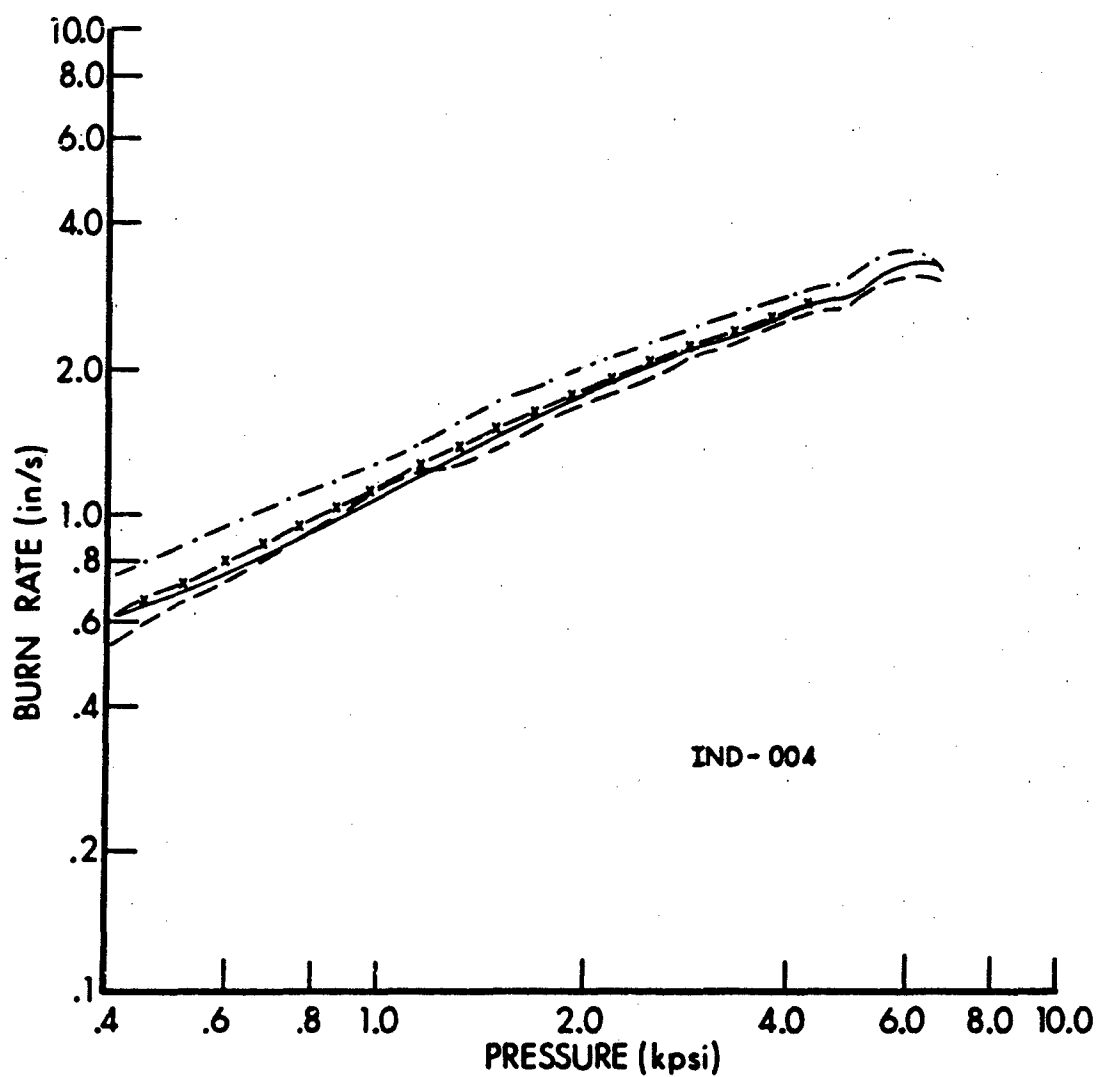


Figure 4. Burning-Rate of IND-004 Black Powder as a Function of Pressure

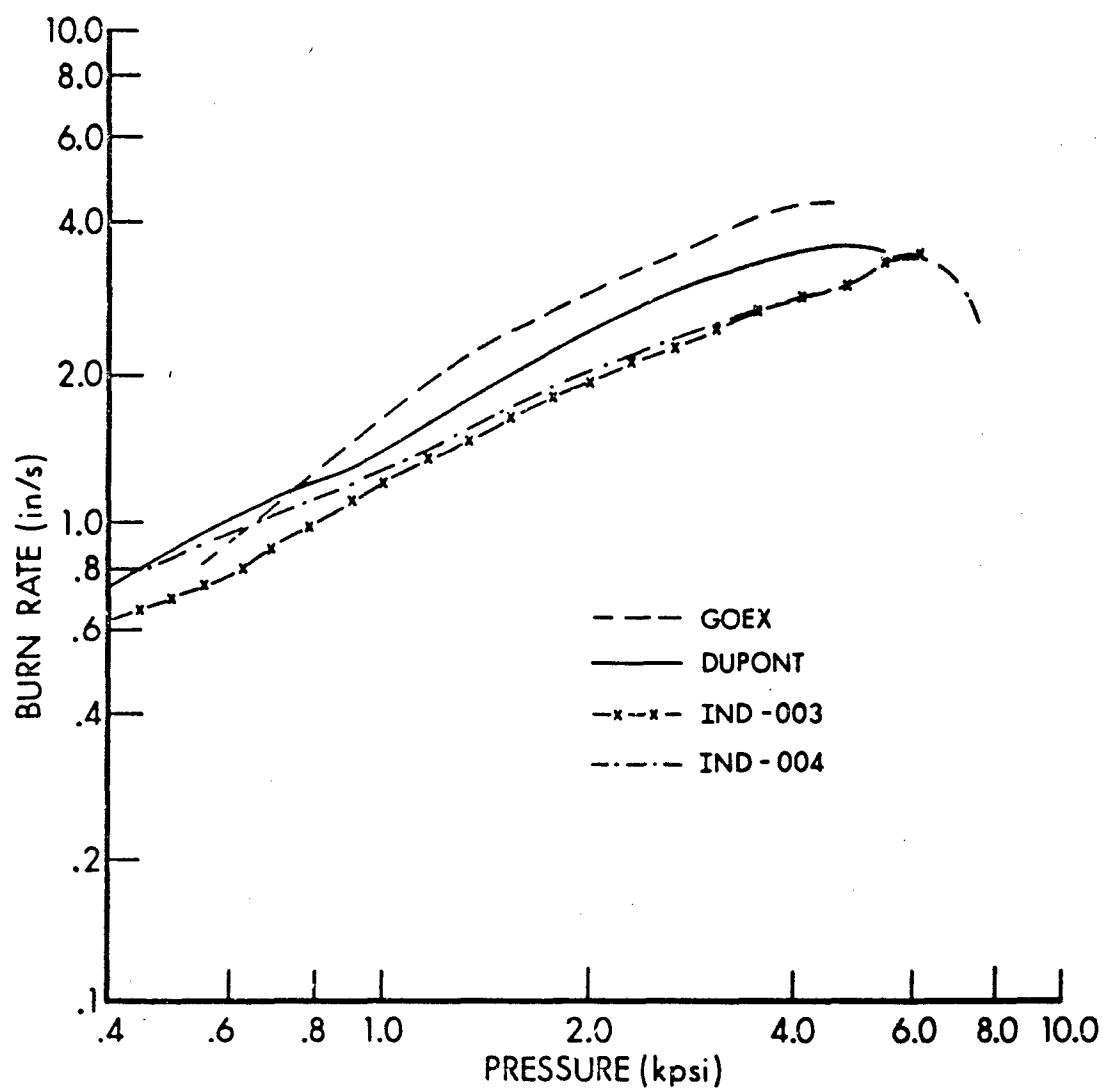


Figure 5. Comparison of Burning-Rate Curves for Black Powder Samples

TABLE 3. Quickness Values and Burning-Rate Equations

Sample Type	Pmax (Kpsi)	Quickness (Mpsi/s)	r=bp ⁿ	
			b	n
GOEX	6.68	0.6539	0.01706	0.671
75-44	6.69	0.6587	0.05202	0.531
	6.68	0.6564	0.01916	0.660
	6.66	0.6708	0.02291	0.641
DuPont	6.89	0.5646	0.05873	0.493
111-12	6.85	0.5003	0.05406	0.485
	6.79	0.4712	0.04658	0.503
	6.83	0.4732	0.02322	0.547
IND-83B	6.74	0.4283	0.01193	0.661
001-003D	6.75	0.4166	0.02756	0.553
	6.80	0.4155	0.02963	0.542
	6.86	0.4276	0.03240	0.533
IND-83B	6.77	0.3986	0.02719	0.547
001-004 D	6.77	0.3839	0.02961	0.577
	6.75	0.4425	0.04044	0.511
	6.72	0.4052	0.03519	0.514

Burning rate r, in inches per second, pressure P in psi and the burning-rate equation is $r = bp^n$

burning-rate equation calculated from data from 2000psi to 4000psi. The narrow pressure range was chosen to avoid the curvature of the data which was most evident in the burning-rate curves for the DuPont and GOEX samples. The ordering of quickness values by propellant type gives the same ordering as does the graphs of the bomb data.

The Indiana samples exhibited similar relative-quickness values and burning-rate equations were similar. No difference was observed that could be attributed to the different degree of acidity of the two samples of potassium nitrate supplied by Croton and Vertac. The Indiana samples burned slightly slower than the DuPont standard but this real difference could be due to the different grain size distributions; on this basis we would expect them to perform well in a gun. It is not clear that the faster burning GOEX samples will perform "better" and only gunfiring results can be used as the criteria for acceptance. With those results we can ascertain the relationship between closed bomb results and gun performance. This analogy to gun performance applies to any propellant-laboratory measured parameter.

B. Grain-Size Distribution

One major concern of this and other closed-bomb black powder work is the large value of the burning-rate exponent, 0.6, in the burning-rate equation as contrasted to the supposedly equivalent value derived from strand burn-rate experiments where a smaller value of 0.164 has been reported by Sasse'.⁹ This difference has been discussed first by Rose¹⁰ and later by White and Sasse'.¹¹ The difference in exponents reflects a different burning mode in the closed bomb than was originally assumed and hence the closed bomb burning rates should be considered as pseudo burning rates.

In the strand-burning experiments, room temperature gas prepressurized the chamber and sample as contrasted to the closed bomb where combustion gases had this effect. It was thought that this different temperature and pressure history might influence combustion to a degree that would change burning-rate exponents. Unpublished data of Sasse' where a single inhibited black powder cylinder was burned in the same closed bomb at pressures to 1600psi, gave an exponent of 0.192, a value near 0.164 derived by cinematography from

⁹(a) R.A. Sasse', "Strand Burn Rates of Black Powder to One Hundred Atmospheres," *Eighth International Pyrotechnics Seminar*, p. 588, IIT Research Institute, Chicago, IL, July 1982. (b) R.A. Sasse', "Strand Burn Rates of Black Powder to One Hundred Atmospheres," *Proceedings of the 19th JANNAF Combustion Meeting*, CPIA Publication No. 366, Vol. 1, p. 13, October 1982.

¹⁰J.E. Rose, "Black Powder-A Modern Commentary-1979," *Proceedings of the 10th Symposium on Explosives and Pyrotechnics*, p. 5a-1, February 1979.

¹¹(a) K. White and R.A. Sasse', "Combustion and Flame Characteristics of Black Powder," *Proceedings of the 18th JANNAF Combustion Meeting*, CPIA Publication No. 347, Vol. 2, p. 253, October 1981. (b) K. White and R.A. Sasse', "Relationship of Combustion Characteristics and Physical Properties of Black Powder," ARBRL-MR-03219, Ballistic Research Laboratory, Aberdeen, MD, November 1982. AD# A122264.

strand-burner experiments. From this relationship it was concluded that the difference in gas temperature and pressure history does not induce a different combustion mode and, furthermore, deconsolidation and/or porous burning does not take place. The large burning-rate exponent must be an artifact of burning a collection of grains of black powder.

To explain this discrepancy one must invoke mechanisms whereby surface area increases during combustion and two substantive suggestions have been offered. One is that grain break-up or fracture is the root cause for increasing surface area¹¹ and a second hypothesis is that all black powder grains do not ignite at the instant of ignition. Either explanation could account for a high value of the exponent, slope, in the burn-rate equation. Another approach to this problem is to consider that the graphite coat acts as an inhibitor and combustion progresses from a single point ignition source that results in increasing surface area during the burn. High-speed photography suggests this effect. If this idea is mechanistically correct, then green grains (where surface burn-rates are greater by a factor of 5 than bulk values) should burn with smaller burn rate exponents than graphited material. Closed-bomb experiments exerting a maximum pressure of 1500psi followed this pattern and the approach seemed promising; however, in the present work, and at pressures to 7000psi, green and graphited grains gave the same combustion curves. The contradiction of the two sets of experiments is unresolved and the concept of single-point ignition is not supported even though this process may be operative. Work in this area should continue for the contradiction is the behavior of black powder.

In dealing with combustion of black powder it is recognized that combustion rates are proportional to grain size and the size distribution should be known. The function was measured for all samples and they are given in Table 4. The grain size distribution for GOEX 75-44 is out of specification, i.e., 11.70% passed through a number 8 sieve while the specification permits a maximum of 5.0%. Moreover, each sample had a slightly different distribution and in all cases the function was not sharp. Under these conditions one worries that the numerous small or large grains dominate the calculation and invalidate the assumption of "average radius." Since an average and particular radius was chosen for data evaluation, it seemed worthwhile to perform a sensitivity analysis with our computer code using different radii. This was accomplished using the set of GOEX data and the various radii of the sieve sizes, four through eight, embracing the sub-sizes of class-one black powder. Results are given in Figure 6 and burn-rate coefficients are given in Table 5. The burn-rate curves have similar exponents, slopes, but they are displaced one from another where the pre-exponent changes by a factor of 2.5. Clearly, the distribution function should be folded into the calculation, but the main point is that assumptions relating to the average radii affect the pre-exponent and not the burning-rate exponent.

C. Density

In characterizing black powder, one would like to assess some measure of its structure. One quality that can be readily obtained is internal-free volume deduced from the difference between mercury and helium densities. Such values are given in Table 6, and they range between four to five percent; similar internal-free volumes were found for other fast burning black

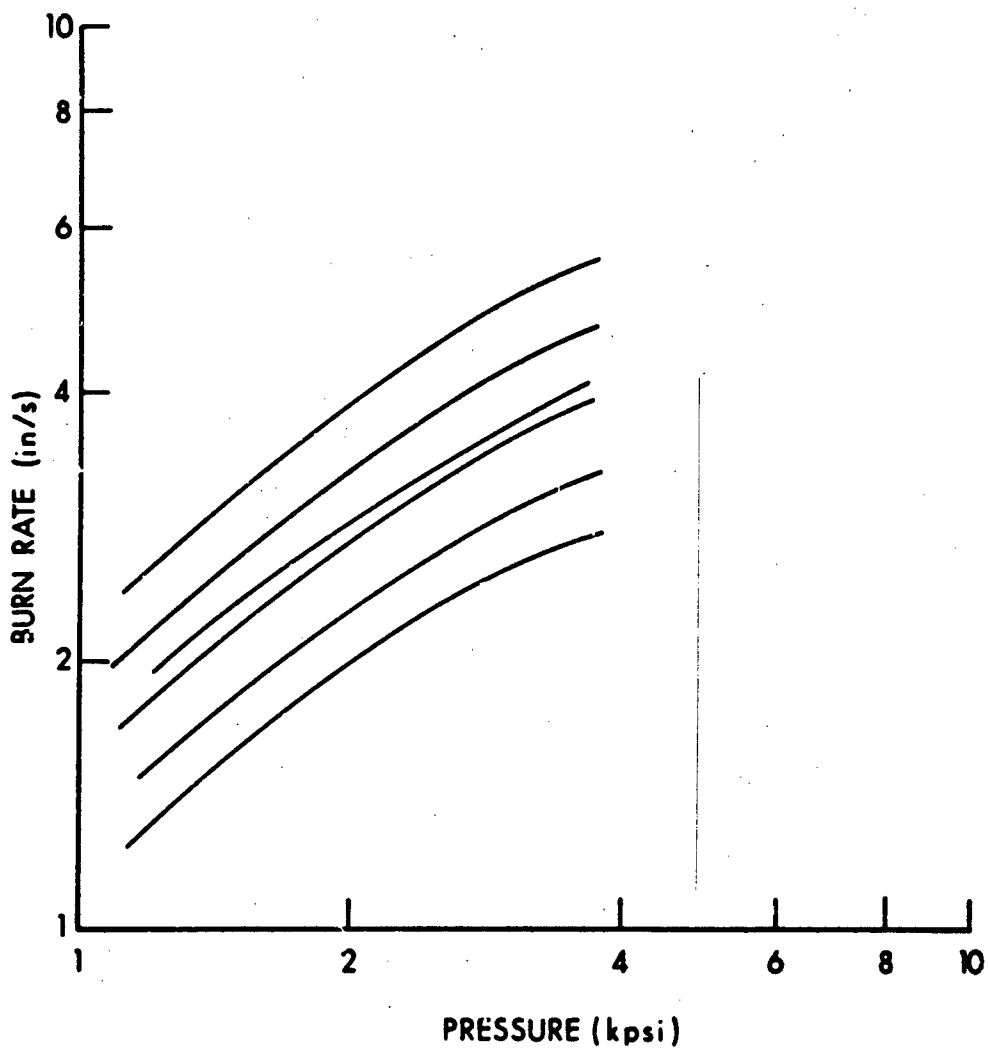


Figure 6. Calculated Burn Rates Assuming Different Average Black Powder Radii r . From Top to Bottom Curve 0.187, 0.157, 0.140, 0.132, 0.110, and 0.094 inch.

TABLE 4. Grain Size Distribution

<u>Sieve#</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u><8</u>	<u>Total %</u>
Largest opening in mm	4.75	4.00	3.35	2.80	2.36		
<u>Lot</u>	<u>Values below in weight percent</u>						
GOEX 75-44	0.31	9.48	26.69	22.25	28.46	11.70	98.89
DuPont 111-12	3.39	33.23	36.13	17.31	8.82	0.65	99.53
IND 83 B 004 C	2.44	22.20	29.80	25.48	18.02	1.53	99.47
IND 83 B 004 D	1.53	24.01	34.35	24.28	14.44	0.90	99.51
IND-83 B 003 C	4.18	27.73	39.10	19.93	7.89	0.85	99.68
IND-83 B 003 D	3.26	20.25	30.23	23.42	19.53	2.73	99.42

TABLE 5. Effect of Using Different Radii for Burn-Rate Equations

<u>Diameter inches</u>	<u>Diameter mm</u>	<u>Burning-Rate Equation Coefficients</u>	
		<u>b</u>	<u>n</u>
0.094	2.39	0.0123	0.662
0.110	2.79	0.0150	0.655
0.132	3.35	0.0172	0.662
0.140	3.56	0.0171	0.671
0.157	3.99	0.0205	0.662
0.187	4.75	0.0244	0.662

Burning rate r , in inches per second,
pressure P in psi and the burning-rate equation is
 $r = bP^n$

TABLE 6. Density and Free Volume of Black Powder

<u>Sample Type</u>	<u>True*</u> <u>Density</u> g/cm ³	<u>Bulk**</u> <u>Density</u> g/cm ³	<u>Internal Free</u> <u>Volume</u> %
DuPont 111-12	1.939 1.933	1.77 1.79	4.50
IND-83 B 004D	1.971 1.972	1.79	5.10
IND-83 B 003C	1.969 1.965	1.77	5.30
IND-83 B 003D	1.968 1.965	1.77	5.30
IND-83 B 004C	1.964 1.962 1.961	1.79	4.10
GOEX 75-44	1.955 1.949	1.75 1.76	5.75

* Measured by Robin J. Davala of BRL-TBD.

** Supplied by ICI Americas, Inc. from data sheets.

powders.¹² Taking the average helium density for charcoal⁸ of 1.45 gm/cm³ together with the densities for potassium nitrate and sulfur, the theoretical density for this black powder is 1.97 gm/cm³, a calculated value equal to measurements given in Table 6. Other data¹² indicate that black powder must be at least as dense as 1.3 gm/cm³ to form a rigid body. The military specified density is 1.74 to 1.80 gm/cm³ and we have not been able to find experiments, or comment, to support this specification; it is possible the density range was selected by the capacity of the press at the time the specification was written.

D. Flame-Spread Rates

A great deal of effort has been devoted to develop a flame-spread "tester" and most of the work was performed by the Princeton Combustion Research Laboratories* of NJ.¹³ The objective was to devise a relatively quick and functionally related test that could be exercised during the production of black powder such that results would be available before material was packaged. It was envisioned that this test would enable one to determine, in a timely manner, if a production cycle produced a ballistically acceptable product and/or would indicate if process or material changes were required. The tester is proposed as a quality assurance technique the importance of which will be discussed by David Hansen in a soon to be published report.¹⁴ Most of the experiments measured black powder's burning rate in a 19cm tube with open slits and using soft igniters that first vented into a small plenum chamber. This is the functional design of the Princeton Laboratory "Flame-Spread Tester." Results obtained by Messina, Ingram, Summerfield and Allen¹³ are given in Table 7 together with some unpublished work done at ICI Americas, Inc., by Smith, Everson, Hansen, and Sasse'.¹⁵ Both data sets are similar; however, the deviations are large. A new version of the tester is proposed by Messina where the cross-sectional area is

¹²(a) R.A. Sasse', "The Influence of Physical Properties of Black Powder on Burning Rate," *Seventh International Pyrotechnics Seminar*, Vol. 2, p. 536, IIT Research Institute, Chicago, IL, July 1980. (b) R.A. Sasse', "The Influence of Physical Properties on Black Powder Combustion," ARBRL-TR-02308, Ballistic Research Laboratory, Aberdeen, MD, March 1981 (AD A100773).

*This is a proto-device developed under MM&TE project number 5764303 and supported by The Product Assurance Directorate, Dover, NJ.

¹³N.A. Messina, L.S. Ingram, M. Summerfield, and J.C. Allen, "Flamespread Propagation Rates of Various Black Powders Using the PCRL-Flamespread Tester," *Seventh International Pyrotechnics Seminar*, Vol. 1, p. 388, IIT Research Institute, Chicago, IL, July 1980.

¹⁴D. Hansen, "Developments in Flame Testing for Continuous Production of Black Powder," DRSMC-QAR-R(D), Artillery Systems Div., Product Assurance Directorate, Dover NJ, 1984 in press.

¹⁵Unpublished work of Dave Hansen (QAR-R), Ronald Sasse' (BRL), Jan Smith and Gil Everson, both of ICI Americas, Inc., June 1983.

TABLE 7. Flame-Spread Rates

Description	Open Air Non-Contained cm/s	Soft Ignition Open Holes cm/s	Soft Ignition Open Slits cm/s	Soft Ignition Closed Holes cm/s	Hard Ignition Closed Holes cm/s
Lot 5*	47 BRL	2138 2740 2469	1041 PR	10363	4100 BRL 11363
Lot 10*	50 BRL	1121 1465 1483	1207 PR	7327	7400 BRL 10026 10762 9670
Lot 11 (GOEX 75-44 with special glaze)	64 BRL	3561 3528 3048	3556 PR	7936	9700 BRL 6145 8581 10026
DUP 11-12	61.3 65.7		1827 IND 1532 IND 1847 IND 1822 IND 1465 IND 1532 IND	10886 9293 7327	
GOEX 75-44	57 BRL 73.0 75.4	4300 3175 3372 3098	3747 PR 3027 IND 3169 IND 2952 IND 3121 INC	12672 7819 11484	5800 BRL
IND-83B 001-003C	70.8 57.5		2270 IND 2281 IND		
IND-83B 001-003D	63.4 65.7		2252 IND 2282 IND 1700 IND		
IND-83B 001-004C	63.9 61.0		2403 IND 2628 IND		
IND-83B 001-004D	61.3 63.0	2817 4444 3200 3419 3125 4348	2329 IND 2463 IND 2891 IND 1883 IND	5443 5773 7056 5443	

 Values with identifiers are the average of three or more values.

*Black powder made from oak charcoal; other black powders made from maple charcoal.

BRL - White, Holmes, and Kelso, rf. 4

PR - Messina, Ingram, Summerfield, and Allen, rf. 13

IND - Hansen, Everson, Smith, and Sasse', rf. 15

increased to reduce wall effects and it is proposed that this revision will result in measurements that have a smaller deviation. David Hansen of the Product Assurance Directorate will have the flame-spread rates of all of the black powders re-evaluated by Princeton Combustion Laboratory using this "improved" flame-spread tester.

The values marked as IND and PR were obtained using photo diodes as combustion indicators and values with no identifiers were obtained using pressure gauges. Either method gave nearly the same result. Pressure wave forms at the front and rear gauges were of the same shape and amplitude in a particular data set. Also, high-speed motion pictures showed the holes becoming luminous at a constant rate and both of these observations indicate a uniform combustion mode. The two Indiana lots gave similar burn rates which are slightly lower than DuPont samples; however, both materials have slower flame-spread rates than GOEX material. This ordering by propellant type is similar to that given by the closed-bomb experiments.

Earlier, White, Holmes, and Kelso⁴ made similar measurements using plugged tubes and brisant igniters, and we repeated these tests using soft ignition. These data sets can be compared in Table 7 and it can be seen that the flame-spread rates in some cases were much faster when using soft electric match igniters. It is proposed that harsh ignition in some of the experiments crushed the grains which in turn restricted flame/pressure propagation. Unfortunately, this latter plugged mode more realistically reflects the true gun environment.

The experiments using plugged holes and harsh ignition as well as some experiments using a plugged tube and soft ignition, showed unusual pressure wave forms within a data set. Some burns resulted in rear pressure spikes that were twice as large as others of the same lot, and motion pictures⁴ revealed that the holes near the ignition source opened more slowly than those at the rear. Both observations lead to the suggestion that in some cases using a plugged tube results in a late thermal explosion. Due to the particular length of the tube and overall burn rates, the slow initial propagation rate was followed by a faster thermal deflagration and these two actions in some cases resulted in nearly the same burn times as was incurred for a smooth burn. Therefore, some of the high burn rates reported for harsh ignition, that appear equal to open-tube values, in fact embrace two different modes of combustion. One could estimate burn times from the pressure wave forms for the DuPont and GOEX samples but the values, in Table 7, for the Indiana samples are highly suspect in that the rear pressure waves were of non-uniform shape and in picking a particular burn time one could be in error by a factor of two.

To complete the study, open-air flame-spread rates were determined and are listed in Table 7 with corresponding data from Reference 4. These measurements do not reflect the large deviations associated with other measures of flame-spread rates but the range of values is small. The ranking of black powders using this test indicates that the Indiana and DuPont powders are similar and have flame-spread rates less than the GOEX material.

E. Strand-Burn Rates

One of the combustion tests invoked was to grind grains of black powder such that it passed through a 120 mesh screen. This material was pressed into sticks which were dried, burned, and photographed using high-speed movie techniques.⁹⁻¹¹ Burning rates were determined from the slope of the position history of the burning interface plotted as a function of time. From previous work the regression error of estimate is about two percent. A sample made from green grains, supplied by Indiana, had a burn rate of 0.88cm/s ($D=1.84$)* at atmospheric pressure, while similar samples made from graphited material from Indiana, DuPont, and GOEX had strand-burn rates of 0.96cm/s ($D=1.717$), 1.05cm/s ($D=1.785$) and 1.09cm/s ($D=1.799$). Expressing these values at the same density,⁹ that $D=1.84$, the burn rates would have been 0.906, 1.00, and 1.01cm/s. The two Indiana samples, graphited and non-graphited, have burn rates within two percent and thus, the presence of graphite is not reflected by this measurement. The ranking of samples shows both DuPont and GOEX burning faster than the Indiana black powder. We realize that only one sample of each type was evaluated and more work is required; however, the data are reported to show that the method is feasible.

In these measurements original grain size or distribution of sizes is negated as is any effect related to the graphite coat or original density of the grains. Under these circumstances, burn rate is only proportional to chemical composition, original fine degree of grinding, and the density of the stick. One advantage of the measurement is that combustion is photographed and one can be certain of the mode of combustion. For all these reasons the test appears to have merit and it parallels the DuPont and GOEX methods where black powder is placed in a lead tube and its diameter is reduced by drawing the sample through an extrusion bench. One difficulty is that material in the lead tube creates internal pressure that is not controlled.

F. Speculation

One result of this evaluation was that the Indiana samples burned at the same speed or slower than DuPont and GOEX reference standards. Considering the jet mill produced a finer grind than the conventional process one would expect equal or faster combustion for the Indiana samples. One of the differences between the two processes is that the older technique used a wheel mill or "incorporating step" to make black powder. It has been claimed that this step forces both sulfur and potassium nitrate into the charcoal pores but S.E.M. micro-photographs do not support this idea. We suggest a different effect. The ground ingredients are damp, about four percent water, some of which wets the surfaces. However, most of the water must be associated in the charcoal much like a sponge absorbs liquid. This model suggests that the four percent bulk water, if just in the charcoal, would make the charcoal 25% in a liquid saturated with potassium nitrate. With the long time periods involved with the wheel-mill step, the charcoal is kneaded, compressed and relaxed, to draw solution into its matrix. This process could result in a faster burning charcoal and account for the particular ranking of black powders found in this study.

* D is density in g/cm^3

These statements do not infer that the Indiana black powder burns too slow. The burning rates are adequate.

IV. CONCLUDING REMARKS

Closed-bomb techniques were used to measure quickness and determine burning-rate equations for two standard class one black powders of DuPont and GOEX and the materials were compared to two lots of black powder made by the Army Ammunition Plant in Indiana that used non-neutral salts of potassium nitrate. These two Indiana lots gave the same combustion results and no effect was attributed to the pH of the salts. The Indiana black powders burned much like DuPont samples; however, all burned slower than GOEX black powder. The fact that the Indiana black powder performed almost as well as one acceptable standard, leads us to expect that both black powders will perform well in the gun ballistic firings. It is not clear that the faster burning GOEX samples will behave differently in ballistic tests.

It was noted that closed-bomb burning-rate equations have larger exponents than are derived from strand-burning rates and it is proposed that the larger exponents embrace both combustion and grain break-up or non-uniform ignition. In contrast, the precision of the strand-burn rates is good and they reflect an uncomplicated and defined mode of combustion i.e., that of a "cigarette burn." Such values could be used to provide a more precise ranking of black powder performance. The ranking of black powders by strand-burn rates is: DuPont and GOEX's burn rates were equal and both were slightly faster than Indiana's black powder.

The variance in flame-spread rates is large and combustion in plugged 105mm primer tubes using soft ignition, at times, leads to large pressure spikes. When brisant igniters are used, grain break-up compacts¹¹ the powder, reducing flame-spread rates. Soft ignition and vented tubes seem to produce a uniform burn. GOEX had faster flame-spread rates than either DuPont or Indiana black powders. Depending on the experimental conditions the relative ranking of Indiana and DuPont material interchanged: for open-air tests DuPont grains burned as fast as Indiana's, for soft-ignition tests and open holes this order reversed, and for plugged holes and soft ignition GOEX and DuPont powder burned equally quickly and Indiana powder gave no meaningful values. Harsh ignition and plugged holes resulted in some cases in restricted flow and this technique cannot be used to rank combustion properties.

This report, which characterizes black powder by various combustion, chemical, and physical tests, will not be complete until it is coupled with gun-firing results expected in 1984. It is the mating of the laboratory and performance data that will establish a technical data base from which to derive functionally related military specifications.

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APPENDIX A
CLOSED-BOMB BURNING RATES

APPENDIX A - Closed-Bomb Burning Rates
Burning Rate, in/s

Pressure Kpsi	GOEX 75-44					DuPont 111-12					IND - 003					IND - 004				
	2.32	2.35	1.93	1.97	1.97	1.97	1.83	1.52	1.68	1.56	1.55	1.53	1.57	1.42	1.36	1.66	1.40	1.79	2.02	2.22
1.5	2.32	2.35	1.93	1.97	1.97	1.97	1.83	1.52	1.68	1.56	1.55	1.53	1.57	1.42	1.36	1.66	1.40	1.79	2.02	2.22
2.0	2.87	2.90	2.74	2.85	2.85	2.43	2.19	2.09	2.02	1.81	1.83	1.82	1.86	1.72	1.65	1.96	1.79	2.02	2.22	2.40
2.5	3.33	3.34	3.37	3.50	3.50	2.79	2.47	2.37	2.33	2.09	2.08	2.05	2.10	1.96	1.88	2.19	2.02	2.22	2.40	2.57
3.0	3.70	3.69	3.82	3.92	3.92	3.06	2.71	2.61	2.60	2.39	2.30	2.27	2.31	2.17	2.09	2.41	2.22	2.40	2.57	2.71
3.5	3.99	3.98	4.17	4.27	4.27	3.26	2.91	2.81	2.80	2.61	2.50	2.46	2.51	2.35	2.28	2.60	2.40	2.57	2.71	2.81
4.0	4.18	4.20	4.43	4.53	4.53	3.46	3.06	2.97	2.94	2.83	2.69	2.65	2.69	2.54	2.46	2.78	2.57	2.71	2.81	2.91
4.5	4.23	4.31	4.58	4.67	4.67	3.57	3.15	3.05	3.02	2.99	2.86	2.82	2.82	2.69	2.59	2.91	2.71	2.81	2.91	3.05
5.0	4.17	4.32	4.59	4.66	4.66	3.55	3.14	3.02	3.01	3.12	2.98	2.94	2.93	2.78	2.72	3.03	2.81	2.91	3.05	3.21
5.5	3.99	4.20	4.46	4.58	4.58	3.54	3.12	2.95	3.03	3.37	3.25	3.18	3.19	3.04	2.93	3.29	3.05	3.21	3.42	3.64
6.0	3.64	3.86	4.12	4.28	4.28	3.38	3.01	2.80	2.91	3.58	3.41	3.39	3.32	3.22	3.06	3.42	3.21	3.42	3.64	3.86

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